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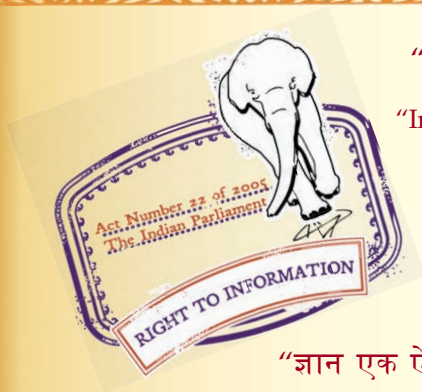
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IS 8590 (1977): Indium sulphate for electroplating [CHD 5: Electroplating Chemicals and Photographic Materials]



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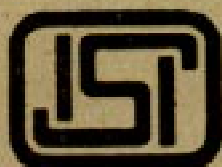


IS : 8590 - 1977

(Reaffirmed 1999)

Indian Standard
**SPECIFICATION FOR
INDIUM SULPHATE FOR ELECTROPLATING**

UDC 661·868·253·2:621·357·7



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Indian Standard

SPECIFICATION FOR INDIUM SULPHATE FOR ELECTROPLATING

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Indian Standard

SPECIFICATION FOR INDIUM SULPHATE FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 17 October 1977, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 Indium is plated on a base metal with a view to providing surface protection, particularly for inhibiting corrosion of cadmium and lead types of bearing alloys. The four most commonly used baths for electroplating indium are of cyanide, sulphate, fluoborate and sulphamate types. Indium sulphate is used in the preparation of sulphate electrolytes and is also supplied in the form of concentrated solution.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for indium sulphate for electroplating.

2. REQUIREMENTS

2.1 Description — Indium sulphate shall be in the form of a white crystalline, deliquescent solid, readily soluble in water, free from foreign matter and visible impurities and shall correspond essentially to the formula $\text{In}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR INDIUM SULPHATE

(Clause 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Solubility in water	To pass the test	A-2
ii)	Indium (as In), percent by mass, <i>Min</i>	33.75	A-3
iii)	Sulphate (as SO ₄), percent by mass, <i>Max</i>	42.38	A-4

3. HANDLING

3.1 Because of its highly hygroscopic nature, indium sulphate should be kept in well-closed bottles. While opening a bottle adequate care should be taken to prevent foreign particles from falling into the material and the closure should be replaced soon after withdrawing the material.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in air-tight containers, preferably with replaceable closure.

4.2 Marking — The containers shall be marked with the following:

- Name of the material;
- Net mass, with mass of equivalent indium (in bracket);
- Name of the manufacturer or his recognized trade-mark, if any; and
- Date and batch number of manufacture to enable the material to be traced from records.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of preparing representative samples of the material and the criteria for its conformity to this specification shall be as prescribed in Appendix B.

APPENDIX A

(Clause 2.2)

METHODS OF TEST FOR INDIUM SULPHATE
FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS:1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. TEST FOR SOLUBILITY IN WATER

A-2.1 Procedure — Weigh accurately about 0.5 g of the material and dissolve in 50 ml of water. The material shall be taken as having satisfied the requirement of the test if there is no turbidity. Reserve the solution for further tests.

A-3. DETERMINATION OF INDIUM

A-3.0 General — Three methods of determination of indium have been given. These are based on: (a) precipitation of indium as indium hydroxide and ignition to indium trioxide, (b) precipitation with 8-hydroxyquinoline, and (c) titrimetric determination using EDTA. In the case of a dispute Method A shall be the referee method.

A-3.1 Method A — Precipitation with Ammonium Hydroxide

A-3.1.1 Reagents

A-3.1.1.1 Ammonium hydroxide — 10 N approximately.

A-3.1.1.2 Ammonium nitrate solution — 2 percent (*m/v*).

A-3.1.1.3 Dilute nitric acid — 1 : 10 (*v/v*).

A-3.1.2 Procedure — Transfer the solution reserved in A-2.1 to a 500-ml volumetric flask and dilute to mark with water.

A-3.1.2.1 Pipette out 200 ml of the above solution into a beaker and heat to boiling. Add ammonium hydroxide until just ammoniacal and boil for 2 minutes. Cool to about 50 to 60°C and stir in a little ashless filter paper pulp.

*Specification for water for general laboratory use (*second revision*).

NOTE 1 — Excess of ammonium hydroxide should be avoided as it may result in low recoveries of indium by peptizing the hydroxide precipitate. The filter paper pulp assists in collecting dispersed indium and in addition, is an aid to filtration.

NOTE 2 — If the solution from which indium is precipitated contains chlorides, especially ammonium chloride, the hydroxide precipitate should be dissolved in dilute nitric acid and re-precipitated and the fresh precipitate washed completely free from chloride, as indium is appreciably volatile in the presence of chloride during ignition.

A-3.1.2.2 Transfer the filter paper together with the precipitate to a tared platinum crucible. Carefully dry the contents, char the paper at as low a heat as possible, burn off the carbon and finally ignite at 750 to 800°C under oxidizing conditions to constant mass.

NOTE — Indium trioxide is readily reduced at high temperature; hence it is necessary to burn off the carbon at as low a heat as possible under good oxidizing conditions.

A-3.1.3 Calculation

$$\text{Indium (as In), percent by mass} = \frac{82.70 M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material in the aliquot taken for the test (A-3.1.2.1).

A-3.2 Method B — Precipitation with 8-Hydroxyquinoline

A-3.2.1 Reagents

A-3.2.1.1 Sodium acetate

A-3.2.1.2 Glacial acetic acid

A-3.2.1.3 8-Hydroxyquinoline solution — Mix 6 g of 8-hydroxyquinoline with 6 g of glacial acetic acid, dissolve in 150 ml of hot water and add ammonia until there is turbidity. Dilute to 200 ml, cool and filter.

A-3.2.2 Procedure — Pipette out 200 ml of the solution prepared in A-3.1.2 into a beaker. Add 2 g of sodium acetate and 2 ml of glacial acetic acid. Heat to 70 to 80°C and add dropwise with stirring 8-hydroxyquinoline solution till precipitation is complete. Cool, stirring from time to time and allow to stand for 2 to 3 hours or for 10 minutes at 40°C. Filter through a tared sintered glass crucible (G No. 4) removing a large part of the precipitant by transferring the precipitate to filter crucible with a small quantity of hot water and then with cold water (25 to 75 ml) until the filtrate is free from the yellow colouration. Dry the precipitate in the filter crucible for 1 to 1.5 hours at 120°C. Cool and weigh to constant mass.

A-3.2.3 Calculation

$$\text{Indium (as In), percent by mass} = \frac{20.99 M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material in the aliquot taken for the test.

A-3.3 Method C — Titrimetric Determination Using EDTA**A-3.3.1 Reagents**

A-3.3.1.1 *Standard EDTA disodium salt solution* — 0.01 M.

A-3.3.1.2 *Sodium potassium tartrate solution* — 5 percent (m/v).

A-3.3.1.3 *Buffer solution* — pH 8-10. Dissolve 13.4 g of ammonium chloride in water. Transfer to a 250-ml volumetric flask, add 88 ml of ammonium hydroxide (R.D. 0.90) and dilute with water up to the mark.

A-3.3.1.4 *Indicator* — Mix eriochrome black T and sodium chloride in the ratio 1:400.

A-3.3.2 Procedure — Pipette out 25 ml of the solution prepared in A-3.1.2 into a beaker. Add 25 ml of sodium potassium tartrate solution followed by 3 to 5 ml of buffer solution until the pH is between 8 and 10. Add indicator until a bright rose colouration appears. Heat to boiling and titrate with standard EDTA solution until the solution turns blue. At the end-point heat again to boiling and wait for 30 seconds; the colour should be stable.

A-3.3.3 Calculation

$$\text{Indium (as In), percent by mass} = \frac{11.48 V \times M}{M_2}$$

where

V = volume in ml of EDTA solution consumed,

M = molarity of EDTA solution, and

M_2 = mass in g of the material in the aliquot taken for the test (A-3.3.2).

A-4. DETERMINATION OF SULPHATE

A-4.0 General — Sulphate is determined gravimetrically as barium sulphate.

A-4.1 Reagents

A-4.1.1 Dilute Hydrochloric Acid — approximately 5 N.

A-4.1.2 Barium Chloride Solution — 10 percent (m/v).

A-4.2 Procedure — Pipette out 200 ml of the solution prepared in **A-3.1.2** into a beaker. Add 25 ml of dilute hydrochloric acid. Heat to boiling, add 20 ml of hot barium chloride solution with stirring and allow to stand overnight. Filter through a tared sintered glass crucible (G No. 4), wash with water and carefully dry at 105°C. Cool and weigh to constant mass.

A-4.3 Calculation

$$\text{Sulphate (as SO}_4\text{)}, \text{ percent by mass} = \frac{41.15 M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material in the aliquot taken for the test (**A-4.2**).

APPENDIX B

(Clause 5.1)

SAMPLING OF INDIUM SULPHATE AND CRITERIA FOR CONFORMITY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing, preparing, storing and handling test samples, precautions given in 3 shall be strictly observed.

B-1.2 The sampling implements and the sample containers shall be clean and dry.

B-2. SCALE OF SAMPLING

B-2.1 In a single consignment all the containers from the same batch of manufacture shall be grouped together to form a lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected from the lot shall depend on the size (N) of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

(Clause B-2.2)

LOT SIZE	SAMPLE SIZE
N	n
(1)	(2)
Up to 10	1
11 to 50	2
51 and above	3

B-2.3 The containers shall be selected at random. In order to ensure randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS: 4905-1968* may be referred. In the absence of random number table, the following procedure may be used:

Starting from any container in the lot, count them as 1, 2, 3,....., etc, up to r and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the sample size). Every r th container thus counted shall be withdrawn so as to constitute the required sample size.

B-3. PREPARATION OF TEST SAMPLES AND NUMBER OF TESTS

B-3.1 From each of the selected containers 6 g of indium sulphate shall be withdrawn. These portions shall be thoroughly mixed to form a composite sample.

B-3.1.1 The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for use as the referee sample. These parts of the composite sample shall be transferred to suitable sample containers and labelled with full particulars for identification.

B-3.1.2 The referee test sample shall bear the seal of the purchaser as well as the supplier. It shall be kept at a place as agreed to between the two, and used in case of any dispute.

B-3.2 Tests for the determination of all characteristics given in Table 1 shall be conducted on the composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For determining the conformity of the lot to this standard all test results on the composite sample shall meet the corresponding requirements given in Table 1.

*Methods for random sampling.

INDIAN STANDARDS
ON
ELECTROPLATING CHEMICALS

IS:

- 330-1968 Chromium trioxide (*first revision*)
- 1809-1969 Nickel salts for electroplating (*first revision*)
- 1880-1977 Zinc oxide and zinc salts for electroplating (*first revision*)
- 2214-1977 Silver nitrate, pure and analytical reagent (*first revision*)
- 3026-1968 Tin salts for electroplating (*first revision*)
- 4846-1968 Sodium potassium tartrate (Rochelle salt)
- 4847-1968 Copper cyanide for electroplating
- 5003-1968 Cadmium oxide for electroplating
- 5761-1970 Gold cyanide and gold potassium cyanide for electroplating
- 6267-1971 Silver cyanide and silver potassium cyanide for electroplating
- 6358-1971 Potassium and sodium cyanides for electroplating
- 6588-1972 Gold chloride (chloroauric acid)
- 7038-1973 Chloroplatinic acid (platinum chloride)
- 7067-1973 Fluoboric acid and metal fluoborates for electroplating
- 8590-1977 Indium sulphate for electroplating